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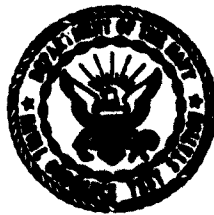
**A STUDY OF THE DECOMPOSITION RATE OF NITRONIUM
PERCHLORATE AT TEMPERATURES OF
80 TO 140°F (26.7 TO 60°C)**

(U)

by

Richard M. Jones
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FOREWORD

During the applied research stage of development of high energy solid propellant systems, formulations containing nitronium perchlorate appeared worthy of investigation.

Studies by other investigators measured the decomposition rates at higher temperatures (over 70°C), and extrapolations were made assuming a temperature independent reaction mechanism.

This study was a two part investigation to determine the amount of decomposition that would be encountered in the long term storage of nitronium perchlorate at ambient temperatures and the decomposition rate of nitronium perchlorate in a fluorocarbon binder system at ambient temperatures.

The study was reviewed for technical accuracy by Dr. Herman F. Cordes. The work was performed under BuWeps Task RMMP-22-028/216-1/F009-06-01.

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ABSTRACT

The rate of decomposition of nitronium perchlorate at constant temperatures of 80 to 140°F (26.7 to 60°C) was determined by measuring the weight loss of modified Kjeldahl flasks from which the accumulating decomposition products were periodically removed. The decomposition rates are based on data accumulated from 550 hours to 28 months. The results indicate a much slower decomposition rate than extrapolations from higher temperature data would indicate and that the incorporation of a fluorocarbon binder had little influence on the decomposition rate. (CONFIDENTIAL).

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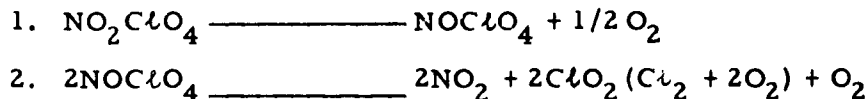
FIG. 1, LHL 092405; FIG. 2, LHL 092617; FIG. 3, LHL 092615;
FIG. 4 through 6, None.

ACKNOWLEDGMENT

The author is indebted to Mr. Edwin L. Moon, former Head of the Properties Analysis Branch, for his assistance in the development of the test method. Special acknowledgment is also due to Dr. Herman F. Cordes of the Research Department for his contribution in evaluating the results of this experiment and to Mr. Bertram O. Stull for fluorocarbon binder samples.

INTRODUCTION

Nitronium perchlorate or nitryl perchlorate (NP or NO_2ClO_4) is an ionic solid that decomposes initially to nitrosonium perchlorate and ultimately to all gaseous products (Ref. 1).



Because of the thermal instability of this oxidizer, studies have been undertaken to determine the problems involved in its storage.

This study was made to determine the decomposition rate of NP in the temperature range of 80 to 140°F (26.7 to 60°C) near ambient pressure and in an atmosphere of its own decomposition products. The temperature range includes temperatures likely to be encountered during storage of this oxidizer prior to its use in solid propellant formulations and during storage of compositions in which it was utilized. Previous studies (Ref. 2) of thermal decomposition were made over the temperature range 158 to 234°F (70 to 112°C). Since NP decomposes into all gaseous products a weight loss method was chosen as a measure of decomposition. No effort was made to identify the decomposition gases or to analyze them quantitatively, as it was beyond the scope of this experiment.

To study the effect of sample size, staggered arrangement of sample weights were used at each test temperature.

To simulate realistic storage conditions, the decomposition was accomplished in an atmosphere of decomposition products.

EXPERIMENTAL

MATERIAL

The NP was obtained from the Callery Chemical Company, Callery, Pennsylvania in about 1 lb lots. The material was stored in refrigerators at about 40°F (5.5°C) prior to testing. The NP was similar to the materials described by Herman F. Cordes in Ref. 2.

APPARATUS

The reaction cells were modified 100 ml Kjeldahl flasks (Fig. 1). The stopcocks were lubricated with Kel F stopcock grease.

A modified D. L. Herring Corporation dry box (Fig. 2), consisting of an inert atmosphere chamber HE-43 and purification train HE-73 was used during sample preparation and weighing operations. A William Ainsworth analytical balance was used for sample weighing in the dry box.

An aneroid recording barometer was used in the dry box to indicate the pressure at the time the reaction cells were opened.

Mechanical convection ovens (Fig. 3) heated with hot water and controlled to $\pm 1.8^{\circ}\text{F}$ (1°C) at less than 30 percent humidity were used for storage of the samples at temperatures above 80°F . The samples stored at 80°F were in the dry box where the room temperature was not accurately controlled. There was a variance of about $\pm 10^{\circ}\text{F}$ in the storage temperature.

SAMPLE PREPARATION

In a dry atmosphere known weights of nitronium perchlorate powder (Test A and B) or NP-fluorocarbon binder strands (Test C), were placed into the modified Kjeldahl flasks. The flasks were sealed and then evacuated and checked for leaks with a spark coil. They were then evacuated further for about 16 hours at about 50 microns.

Test A

In order to obtain the decomposition gases for the test conditions the pre-evacuated flasks were placed in a dry box and preheated in a Kel F oil bath at approximately 212°F (100°C). At this temperature a sufficient quantity of oxidizer was rapidly decomposed to fill the flasks with decomposition products. The flasks were then cooled to ambient temperature and opened in the dry atmosphere at 955 millibars pressure to release the excess pressure and establish an ambient pressure in the flasks. Because of the differential in molecular weight between the gaseous decomposition products and the dry box atmosphere, it was found necessary to keep the same pressure in the dry box at each weighing when exhausting the flasks as small pressure variations would cause noticeable variation in the weight losses.

The flasks were stored in ovens (Fig. 3) at temperatures of 140°F (60°C) and 120°F (43.3°C), inside the dry box at $80 \pm 5^{\circ}\text{F}$ and in the antechamber of the dry box at ambient humidity at $80 \pm 5^{\circ}\text{F}$ (26.7°C). There were two sample flasks and an empty control flask at each of the test conditions.

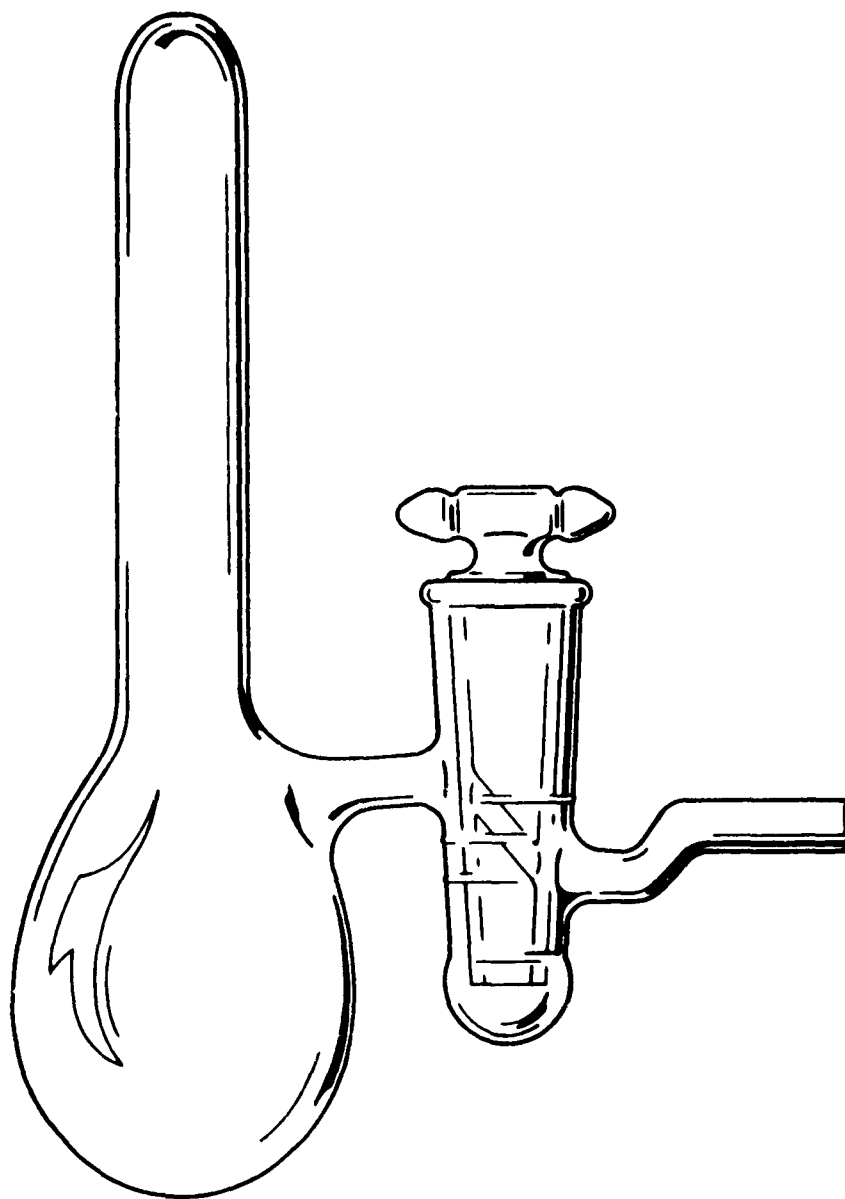


FIG. 1. Modified 100 ml Kjeldahl Flask.

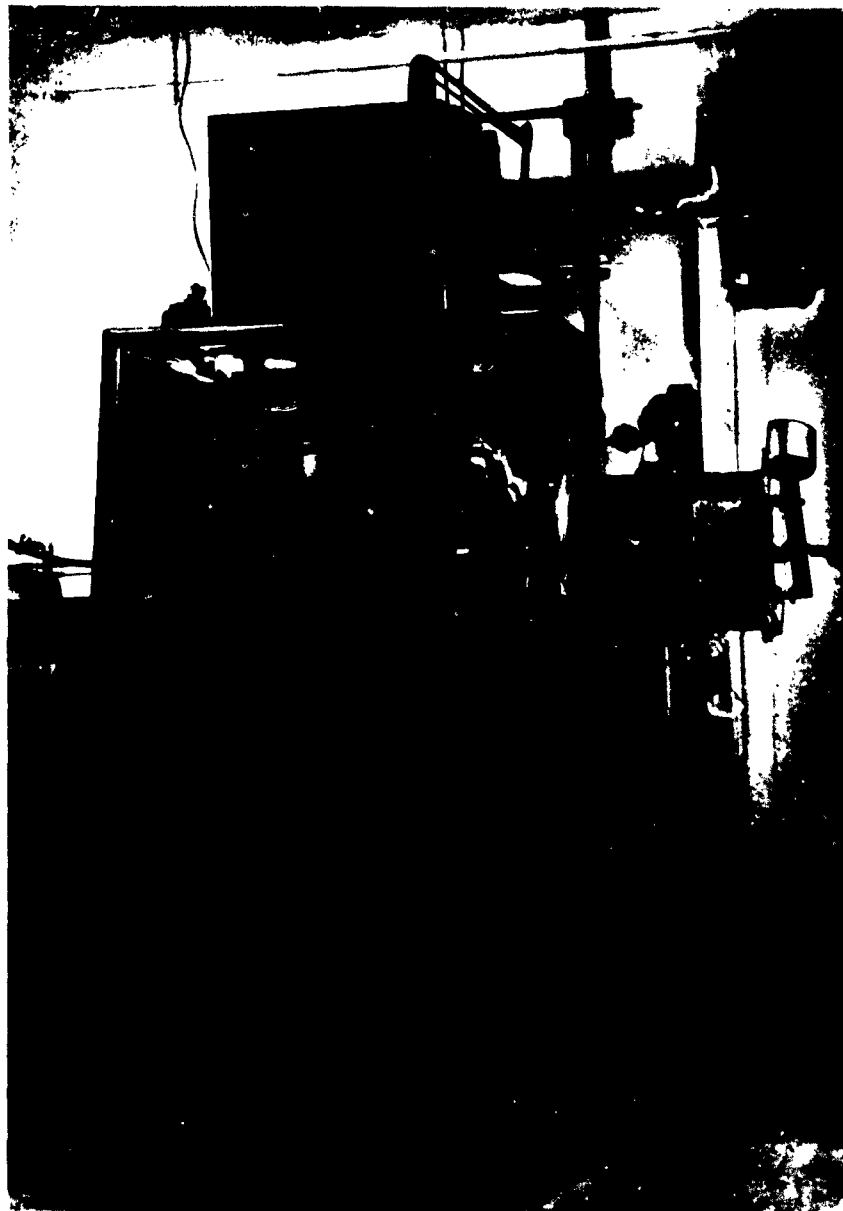


FIG. 2. Modified D. L. Herring Corporation
Dry Box.

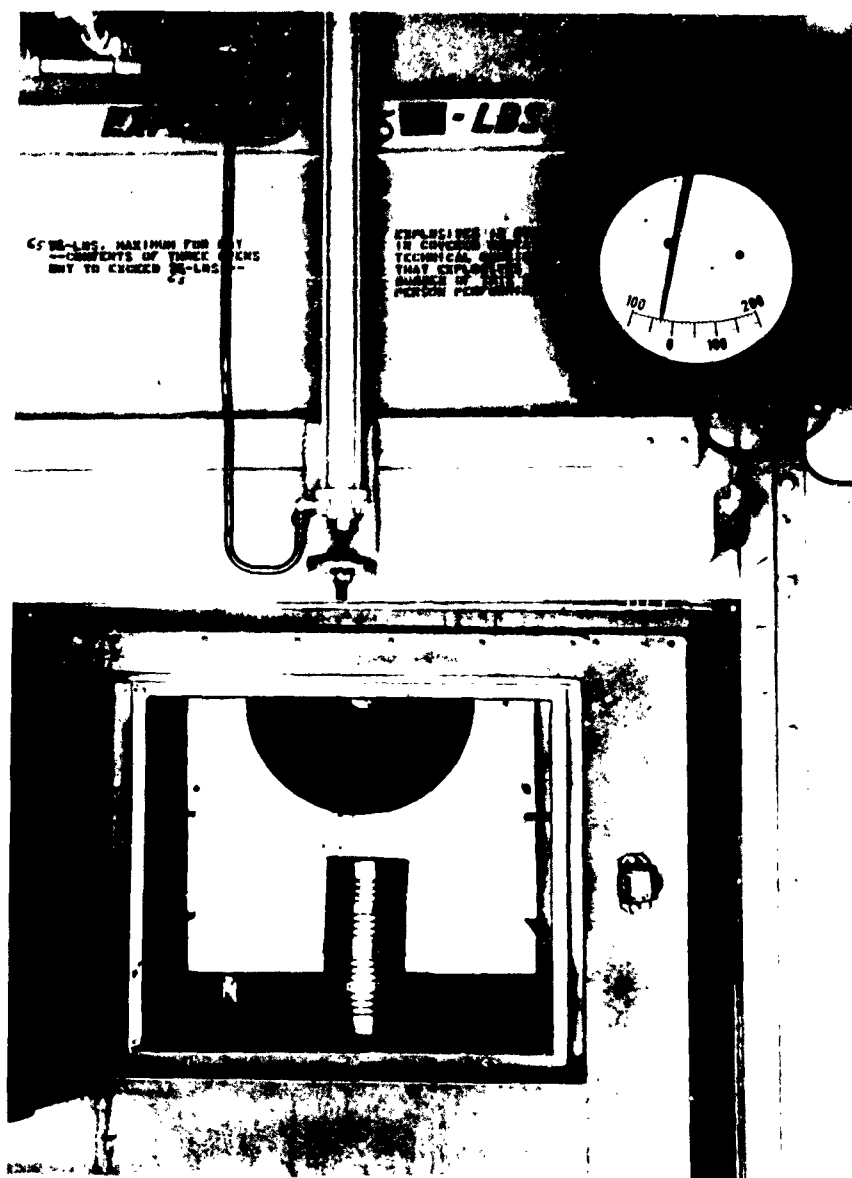


FIG. 3. Mechanical Convection Oven.

Procedure. The flasks were removed from the test conditions at intervals of 2 to 3 days and placed in the dry box. The dry box pressure was then adjusted to, and maintained at, 955 millibars. The flasks were then weighed, opened to dry box pressure (exhausted), closed, reweighed, and placed back into test conditions. Test results are shown in Table 1 and Fig. 4. An ionizing unit was run over the flasks to remove static electricity prior to each weighing.

TABLE 1. Test A Results
(Preheated above 100°C)

Temp, °F	Sample wt, g	Time to 0.1% decomposition interpolated value, hr	Average % decomposition/hr x 10 ²
80 ± 10 ^a	5	23	0.44
	10	19	0.53
	20	58	0.17
80 ± 10 ^b	20	32	0.31
	40	83	0.12
120	10	2.02	4.95
	20	2.04	4.90
140	0.5	0.85 ^c 0.65 ^d	11.80 ^c 15.38 ^d
	2	0.63 ^c 0.55 ^d	15.87 ^c 18.18 ^d

^aSamples stored in dry box.

^bSamples stored in antechamber of dry box.

^cFigures based on total test results.

^dFigures based on equilibrium rate (linear portion of curve).

Test B

In Test B the evacuated flasks were placed in the dry box and decomposition products gases were passed into the flasks from a nitrogen perchlorate source heated to about 212°F (100°C). Therefore, no preheating of the test samples was required. The flasks were opened in the dry box to 955 millibars pressure, closed, and placed in ovens at temperatures of 130°F (54.5°C) and 110°F (43.3°C) inside the dry box and in the dry box antechamber at 80 ± 5°F (26.7°C). There were again two flasks and a control flask at each test condition.

Procedure. The flasks were removed from the test condition at intervals of 3 to 4 days. However, for the flasks stored at 80°F (26.7°C), this interval was increased after about 700 hours in test to an interval of 1 to 4 months. The flasks were weighed, opened to 955 millibars pressure, closed, reweighed, and placed back in test conditions. Test results are shown in Table 2 and Fig. 4. An ionizing unit was run over the flask to remove static electricity prior to each weighing.

TABLE 2. Test B Results
(No Preheating)

Temp, °F	Sample wt, g	Time to 0.1% decomposition interpolated value, hr	Average % decomposition/hr x 10 ²
80 ± 10 ^a	20	50	0.20
	20	185	0.05
80 ± 10 ^b	20	556	0.02
110 ± 2	10	8.8	1.14
	20	10.63	0.94
130 ± 2	5	1.34	7.46
	10	1.09	9.17
80 ± 10 ^c	20	4.5 days	0.0926
	20	7 months	0.0020
80 ± 10 ^d	20	11 months	0.0013

^aSample stored in dry box.

^bSample stored in antechamber of dry box.

^cContinuous storage in dry box for over 28 months.

^dContinuous storage in antechamber of dry box for over 28 months.

Test C

The NP-fluorocarbon binder samples were prepared by extrusion into 1/4-inch diameter strands from about an 80 percent NP-fluorocarbon binder mixture. A composition analysis showed a variation of from 75 to 80 percent NP content in the extruded material. The fluorocarbon composition was 60 percent Viton A and 40 percent Teflon 7.

Based on the composition analysis, a 20 gram sample size was used at 140°F storage and 40 gram sample sizes were used at the other storage temperatures, giving an NP content of approximately 15 and 30 grams, respectively.

The samples were stored at temperatures of 140°F (60.0°C), 120°F (49.0°C), 100°F (37.8°C), 90°F (32.2°C), and 80°F (26.7°C). The preparation and weighing of the samples were the same as in Test B.

Procedure. The flasks were removed from the test conditions at intervals of 1 to 4 days at 140 and 120° temperatures, while intervals of 2 weeks to 4 months were used for the other storage temperatures.

As the samples were removed from the test, they were analyzed for the amount of binder from which the original NP content was calculated. This was accomplished by cutting the individual samples into small pieces, placing them in water for 2 hours decanting and replacing in fresh water overnight to react the NP. The samples were then filtered, dried and weighed. This procedure was repeated until a constant weight was obtained.

The decomposition results are shown in Table 3 and in Fig. 4 and 5.

TABLE 3. Test C Results
(NP-Fluorocarbon Binder)

Temp, °F	NP Sample wt, g	Time to 0.1% decomposition interpolated value, hr	Average % decomposition/hr x 10 ²
80 ± 10 ^a	30.24	8,350 (12 months)	0.0012
	30.48	12,200 (17 months)	0.0008
90	29.79	3,400 (4.7 months)	0.0029
	29.81	2,700 (4 months)	0.0037
100	29.91	2,400 (3.3 months)	0.0042
120	29.53	2.6	3.85
	29.85	2.2	4.55
140	15.18	1.1	9.09
	15.54	1.1	9.09

^a Samples stored in dry box antechamber.

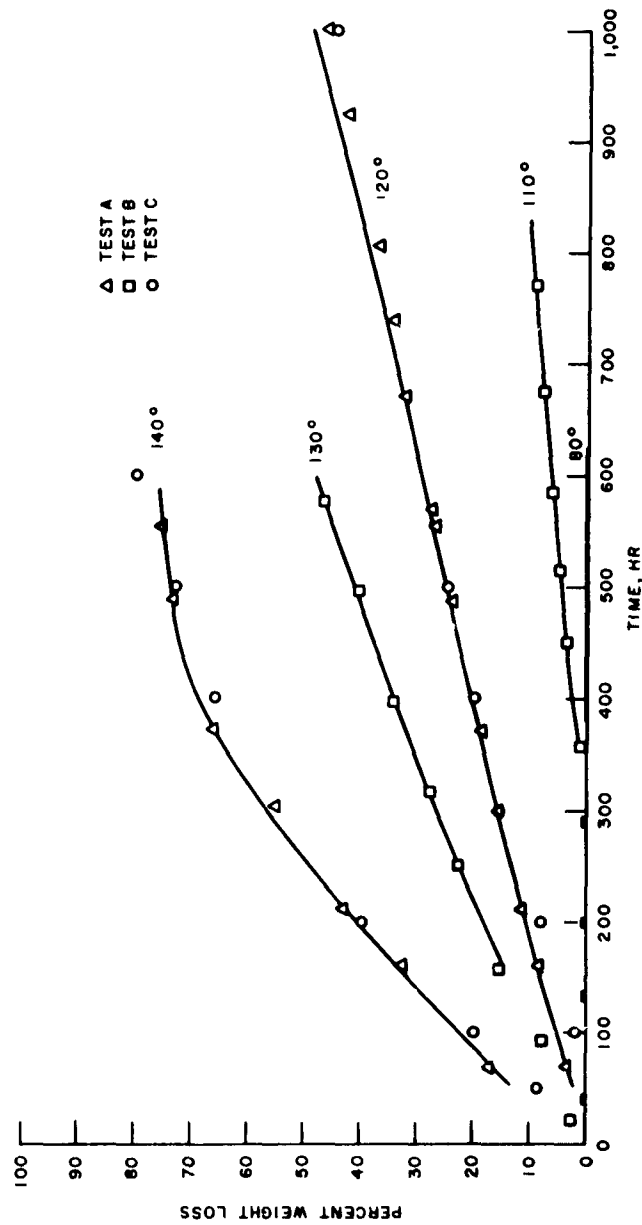


FIG. 4. Average Percent Weight Loss vs Time (Test A, B, C).

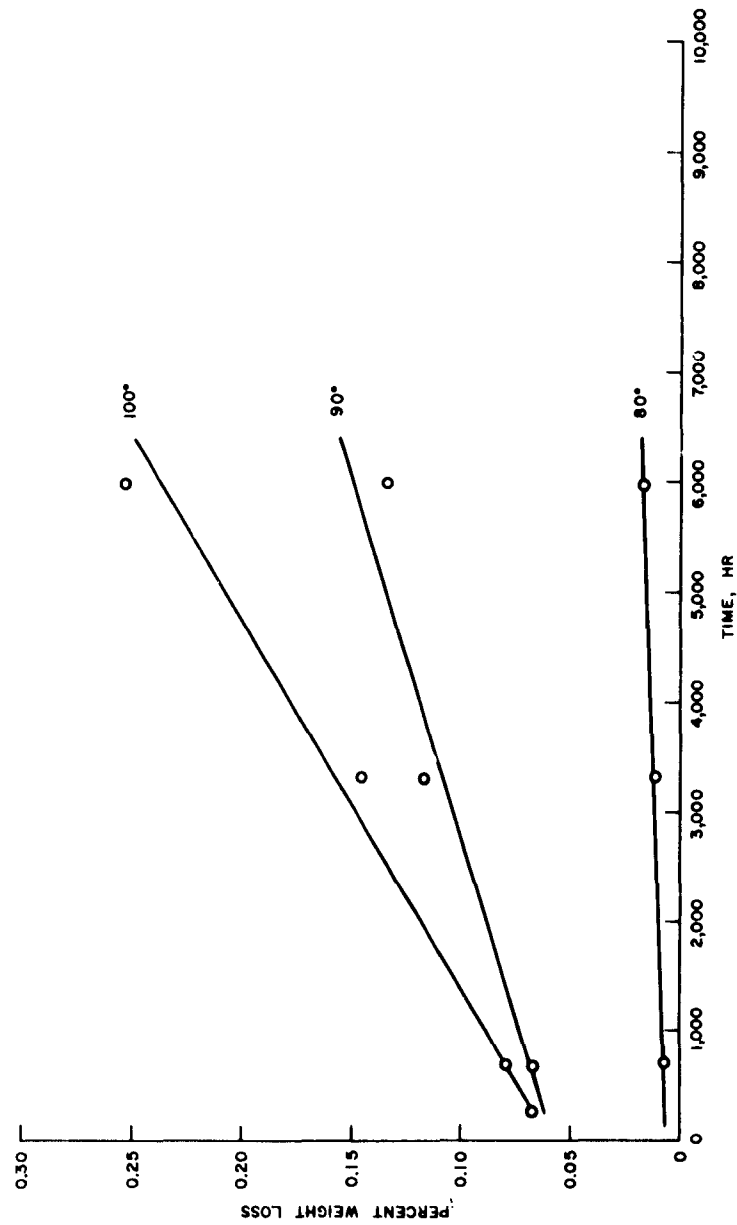


FIG. 5. Average Percent Weight Loss vs Time (Test C-80 to 100°F).

DISCUSSION

The results of this experiment show a conformity to extrapolated values at temperatures of 120°F and above. At temperatures of 110°F, there is a measured decrease in the decomposition rate from the extrapolated value by a factor of about 2, or by a factor of about 160 at 80°F (Fig. 6).

The decomposition rate data was taken from the linear portion of the weight loss versus time curve which was considered to be the equilibrium value. These slopes were measured and plotted versus the reciprocal of the absolute temperature (Fig. 6).

When plotting percent weight loss versus time (Fig. 4), the decomposition rate appears linear up to about 60 percent decomposition at 140°F and to about 50 percent at 130°F. At these percentages of decomposition, there is a slowing down of the decomposition rate. This condition is accounted for by reduction of the amount of NP present in the test.

During preparation of the test samples some of the test samples must have been contaminated, as individual samples have a much higher decomposition rate than their companion samples. All rates, however, were linear and the lower rates were considered more reliable. The results of 80°F storage showed the greatest variation of data of any storage temperatures.

In this experiment, the size of the sample did not appear to have any consistent effect on the decomposition rate. At higher temperatures the larger size samples had the greatest decomposition rates but at temperatures below 120° the reverse was true. Thiokol (Ref. 3) did; however, show some effect of sample size on decomposition rate, but at higher temperatures and smaller sample size.

The activation energies were determined by the mathematical analysis of the Arrhenius plot of the data at temperatures of 80 to 140°F (Table 4).

The formulas used for the calculations of specific reaction rate (K) and activation energy (E) were from Ref. 3 and 4 and are as follows:

$$K = \frac{2.303 \log a^{\circ}/a}{t}$$

TABLE 4. Isothermal Decomposition of NO_2ClO_4

Temp °F	Test	(°K ⁻¹)	K value ^a	Initial wt, g	Remaining wt, g	Decomposed wt, g
140	A	300	6.3 x 10 ⁻⁷	0.50	0.31	0.19
			8.6 x 10 ⁻⁷	2.00	1.04	0.96
			7.5 x 10 ⁻⁷	average		
	C		7.42 x 10 ⁻⁷	15.18	8.15	7.03
			7.29 x 10 ⁻⁷	15.54	8.43	7.11
			7.35 x 10 ⁻⁷	average		
130	B	306	2.0 x 10 ⁻⁷	5.02	3.60	1.42
			3.5 x 10 ⁻⁷	10.00	6.01	3.99
			2.7 x 10 ⁻⁷	average		
120	A	311	1.57 x 10 ⁻⁷	10.00	7.59	2.41
			1.55 x 10 ⁻⁷	20.00	15.21	4.79
			1.56 x 10 ⁻⁷	average		
	C		1.5 x 10 ⁻⁷	29.53	23.80	5.73
			1.7 x 10 ⁻⁷	29.85	23.31	6.54
			1.6 x 10 ⁻⁷	average		
110	B	316	3.6 x 10 ⁻⁸	10.00	9.06	0.94
			3.77 x 10 ⁻⁸	20.00	18.02	1.98
			3.68 x 10 ⁻⁸	average		
100	C	322	1.25 x 10 ⁻¹⁰	29.91	29.83	0.08
90	C	327	6.0 x 10 ⁻¹¹	29.79	29.75	0.04
			8.4 x 10 ⁻¹¹	29.81	29.76	0.05
			7.2 x 10 ⁻¹¹	average		
80	B	333	3.7 x 10 ⁻¹¹	20.00	19.95	0.05
			5.7 x 10 ⁻¹¹	20.02	19.94	0.08
			4.7 x 10 ⁻¹¹	average		
80	C	333	1.4 x 10 ⁻¹¹	30.48	30.47	0.01
			3.3 x 10 ⁻¹¹	30.24	30.22	0.02
			2.3 x 10 ⁻¹¹	average		

^a Average K values were used in the Arrhenius plot (Fig. 6).

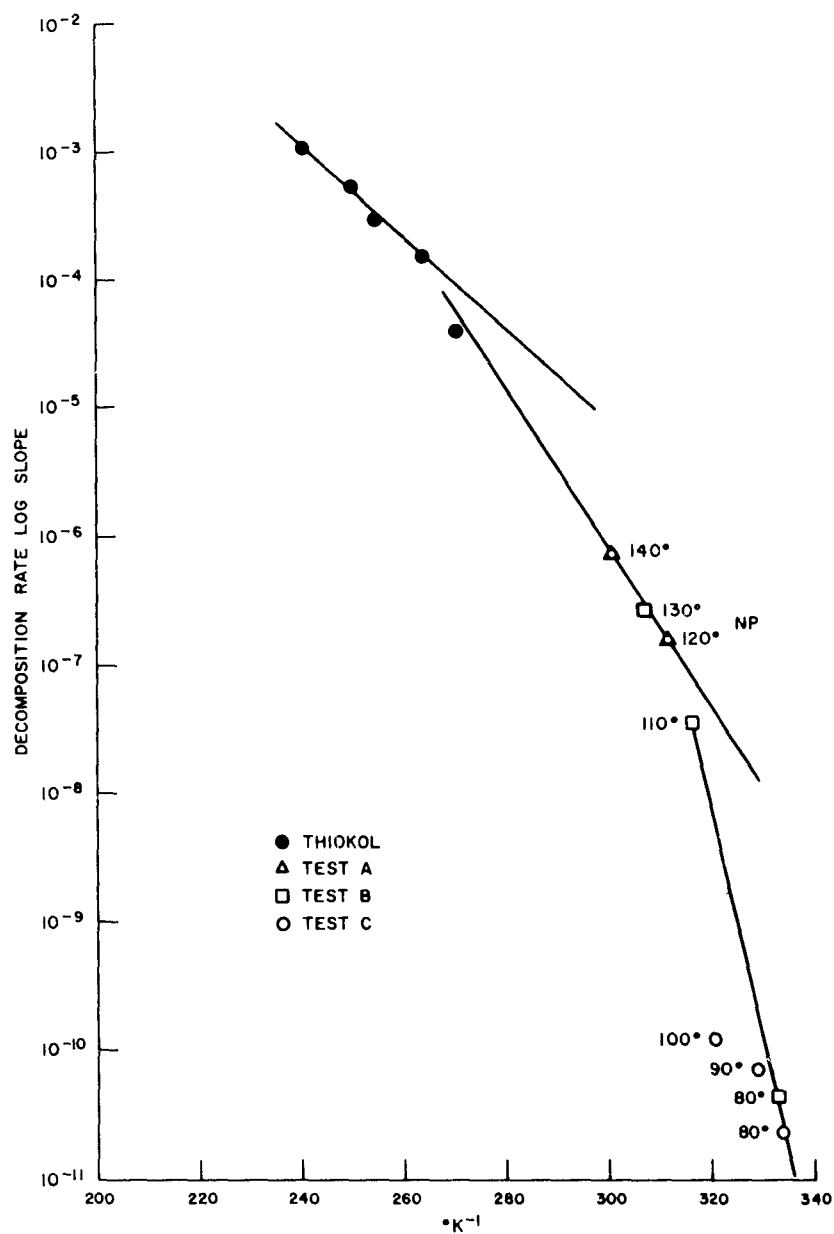


FIG. 6. Arrhenius Plot for Thermal Decomposition of NO_2ClO_4 .

where:

a° = original weight NP
 a = remaining weight NP
 t = in seconds

$$E = \frac{R [\Delta \ln K] 2.303}{\Delta 1/^{\circ}K}$$

where:

$^{\circ}K$ = absolute temperature
 $[\Delta \ln K]$ = slope of plot of $\ln K$
 R = gas constant 1.987 calories per degree per mole
2.303 = conversion of natural log to common log.

Three activation energies are shown by data obtained by Thiokol (Ref. 3), Dr. H. F. Cordes (Ref. 2) and the author. An activation energy of 17 KCal/mole is obtained at temperatures of 205 to 365°F (96 to 155°C), 27.0 KCal/mole at temperatures of 110 to 205°F (43 to 96°C), and 79.0 KCal/mole at temperatures of 80 to 110°F (27 to 43°C). Dr. H. F. Cordes found an activation energy value of 27.49 ± 0.93 KCal/mole at temperatures of 158 to 234°F (70 to 112°C). This would appear to indicate a change in decomposition mechanism in different temperature ranges.

During this experiment no attempt was made to determine the effect of pressure on the decomposition rate of NP.

There appears to be a relation between the interval between weighings and the decomposition rate in that the longer the interval between weighing the lower the decomposition rate measured. This could be due to an equilibrium process between the original atmosphere of the added decomposition products and the initial decomposition product of the sample.

The material did not appear to decrease in volume in the flasks even though over 60 percent decomposition had occurred.

A nuclear magnetic resonance (NMR) analysis was performed on the three samples stored at 80°F. Two samples showed no moisture contamination while one sample showed 0.85 percent per gram moisture. The two samples showing no moisture contamination had 18 percent decomposition and 0.27 percent decomposition respectively. while the moisture contaminated sample had 0.41 percent decomposition. The limit of detection of water in D_2O is about 0.005 percent.

An emission spectrographic analysis was also performed in an attempt to find a contamination producing the high decomposition in one of the samples. The results showed traces of various metals but since all samples showed these emissions, they were not thought to produce the high decomposition rates. It must be assumed that some organic contamination had occurred.

When the NP-fluorocarbon binder samples were removed from test they appeared to be the same size and color as the original samples, however, they had an irregular surface, tended to adhere to each other, and were softer. These conditions became more in evidence as the storage temperatures increased.

The decomposition gas added to and produced in the flasks did not remain dark except in the flasks stored at higher temperatures and on cooling these gases became lighter. This is probably the result of the equilibrium gas from $N_2O_4 \rightleftharpoons 2NO_2$ from colorless to reddish brown color more intense with heat.

No tests were made to determine the ratio of nitronium to nitrosonium perchlorate at completion of the experiment.

CONCLUSIONS

The number of samples and the temperatures involved in the test were small and the data sparse; the data that was obtained did give an indication of decomposition rates which agreed with results obtained by other experimental work which became available while the tests were in progress (Ref. 1, 2, and 3).

NP has a much slower decomposition rate at 110°F and below than is calculated from extrapolation of thermal stability data at higher temperatures.

Although some data by other experimental methods show an effect of sample size on decomposition rate, in this experiment at these temperatures and sample sizes the results are not indicative of an effect of sample size on the decomposition rate (Ref. 3).

It appears, from the experimental data by this and other studies, that the decomposition rate at temperatures of 80°F and below is quite low, and averages showed about 0.1 percent decomposition in 7 to 17 months. It also appears that the purity of the sample as well as temperature have a great effect on the decomposition rate. There also may be a change of mechanism of decomposition at different temperatures. It appears from the data that a pure sample could possibly be stored for a considerable time if the temperature was kept below 80°F. The samples in storage should be checked periodically for the accumulation of gaseous decomposition products and, if found, the sample should be removed from storage.

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